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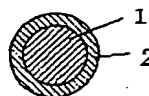
(54)【発明の名称】 被覆蛍光体の製造方法

(57)【要約】

【目的】 水酸基の含有量が少なくして遮水性に優れるシリカコーティング膜を蛍光体の熱等による劣化を防止しつつ形成できて、輝度や発光寿命、ないしその維持性に優れる被覆蛍光体を得ること。

【構成】 蛍光体の粉末(1)を被覆する水酸基含有のシリカ膜(2)中に、一般式： F_nSiR_{3-n} 。(ただし、Rはアルキル基又はアルコキシ基、nは1～3の整数である。)で表されるフッ素含有有機シランを拡散させてシリカ膜と低温で脱水縮合反応させる被覆蛍光体の製造方法。

【効果】 フッ素による撥水性を有する緻密なシリカコーティング膜を形成できて水分劣化が生じにくいシリカ被覆蛍光体を得られる。



【特許請求の範囲】

【請求項1】 蛍光体の粉末を被覆する水酸基含有のシリカ膜中に、一般式： F_nSiR_{4-n} （ただし、Rはアルキル基又はアルコキシ基、nは1～3の整数である。）で表されるフッ素含有有機シランを拡散させてシリカ膜と低温で脱水縮合反応させることを特徴とする被覆蛍光体の製造方法。

【発明の詳細な説明】

【0001】

【産業上の利用分野】本発明は、蛍光体の劣化を防止した被覆蛍光体の製造方法に関し、得られた被覆蛍光体は輝度や発光寿命、その維持性に優れて照明装置や表示装置等の発光型装置などに好ましく用いうる。

【0002】

【従来の技術】従来、蛍光ランプやEL発光体等の照明装置、電子装置用観察スクリーン等の表示装置などにおける発光部の形成に用いる蛍光体は、水分で劣化して発光力や輝度が低下することからシリカ膜で被覆する対策が採られており、かかる被覆蛍光体の製造方法としてCVD方式やゾル・ゲル方式でシリカコーティング膜を形成する方法が知られていた（特開昭61-23678号公報）。

【0003】しかしながら、シリカコーティング膜形成時の加熱温度を高くすると蛍光体が熱劣化してコーティング膜で被覆する前よりも輝度等が低下し、劣化速度も速くなって寿命がより短縮化される問題点があった。一方、蛍光体の熱劣化防止のため200～400℃でシリカコーティング膜を形成すると遮水性に劣るためか水分で劣化しやすい被覆蛍光体となる問題点があった。

【0004】

【発明が解決しようとする課題】本発明は、前記低温形成のシリカコーティング膜にあっては水酸基の含有量が多くて水分が浸透しやすくそのため遮水性に乏しいことを究明して得た知見をもとに、水酸基の含有量が少なく遮水性に優れるシリカコーティング膜を蛍光体の熱等による劣化を防止しつつ形成できて、輝度や発光寿命、ないしその維持性に優れる被覆蛍光体を得られる製造方法の開発を課題とする。

【0005】

【課題を解決するための手段】本発明は、蛍光体の粉末を被覆する水酸基含有のシリカ膜中に、一般式： F_nSiR_{4-n} （ただし、Rはアルキル基又はアルコキシ基、nは1～3の整数である。）で表されるフッ素含有有機シランを拡散させてシリカ膜と低温で脱水縮合反応させることを特徴とする被覆蛍光体の製造方法を提供するものである。

【0006】

【作用】前記一般式で表されるフッ素含有有機シランを水酸基含有のシリカ膜中に拡散させることにより、そのフッ素含有有機シランがシリカ膜中の水酸基を介し室温

等の低温で脱水縮合反応してシリカ膜中の水酸基を消費除去し、かつシリカ膜の縮合反応を促進して緻密なシリカ膜に改質する。しかもフッ素含有のシリカ膜に改質して撥水性に優れるシリカ膜を形成する。

【0007】前記の結果、蛍光体の粉末を熱劣化させることなく遮水性に優れるシリカコーティング膜を形成でき、水分劣化が生じにくくて輝度や発光寿命、ないしその維持性に優れる被覆蛍光体を得ることができる。

【0008】

【実施例】本発明の製造方法は、一般式： F_nSiR_{4-n} （ただし、Rはアルキル基又はアルコキシ基、nは1～3の整数である。）で表されるフッ素含有有機シランを蛍光体粉末を被覆する水酸基含有のシリカ膜中に拡散させてそのシリカ膜と低温で脱水縮合反応させることにより被覆蛍光体を得るものである。

【0009】本発明において用いられるフッ素含有有機シランは、水分ないし水酸基との反応を介して脱水縮合の反応機構を示すものであればよい。一般には、有機基（R）に基づく反応生成物がシリカ膜中に残存しないことが好ましいことから、前記一般式におけるRがメチル基、エチル基、プロピル基、ブチル基の如きアルキル基、又はメトキシ基、エトキシ基、プロポキシ基、ブトキシ基の如きアルコキシ基であるものなどが用いられる。

【0010】フッ素含有有機シランのシリカ膜中への拡散は、適宜な方式で行ってよい。一般にはフッ素含有有機シランの気流を、必要に応じヘリウムやアルゴン等の不活性ガスを介しシリカ膜に対して供給する方式が採られるが、液状のフッ素含有有機シランにシリカ膜で被覆した蛍光体粉末を浸漬してもよい。

【0011】シリカ膜中に拡散させたフッ素含有有機シランの脱水縮合反応に際しては、蛍光体粉末が熱劣化しない温度、一般には200～400℃に加熱してもよいが、好ましい方式は室温等の低温での反応である。

【0012】本発明においてフッ素含有有機シランで処理する対象の、蛍光体粉末を被覆する水酸基含有のシリカ膜については特に限定はない。ちなみに SiO_2 のほか、例えば TiO_2-SiO_2 、 ZrO_2-SiO_2 などの複合形態で形成されたものであってもよい。

【0013】従って前記のシリカ膜は、適宜な方式で形成したものであってよい。一般には、400℃以下での低温CVD方式、減圧CVD方式、プラズマCVD方式、低温ゾル・ゲル方式などの水酸基を含有しやすい方式で形成されたシリカ膜が対象とされる。処理対象のシリカ膜の厚さは、通例30μm以下、就中10nm～1μm程度であるが、これに限定されない。

【0014】シリカ膜で被覆された蛍光体粉末そのものについては特に限定はない。一般には、硫化亜鉛や硫化カドミウム亜鉛を銅、マンガン、アルミニウム、銀、塩素、ホウ素等で活性化したものや、希土類賦活酸化イッ

トリウム等の酸化物などからなる。蛍光体粉末の粒径は任意であるが、平均粒径に基づき $1\mu\text{m}$ 以上、就中 $5\sim 50\mu\text{m}$ が一般的である。

【0015】本発明において用いる、水酸基含有のシリカ膜で被覆された蛍光体粉末は、図1に例示の如くシリカ膜のみで被覆されたものであってもよいし、図2に例示の如くシリカ膜の内部に1層又は2層以上の他のコーティング膜を有するものであってもよい。図中の1が蛍光体粉末、2がシリカ膜、3が他のコーティング膜である。

【0016】前記したシリカ膜以外のコーティング膜の種類は任意であるが、輝度の向上に有効なものとしては高誘電体からなるコーティング膜などがあげられる。その高誘電体の例としては、 Ta_2O_5 、 Al_2O_3 、 ZrO_2 、 TiO_2 、 BaTiO_3 、 PbTiO_3 、 PZT (PbZrO_3 と PbTiO_3 の固溶体)、 PLZT (PZT の La 添加物)、 SrTiO_3 等の高誘電率で透光性の金属酸化物系化合物などがあげられる。

【0017】前記の高誘電体層をシリカ膜の内部に有する被覆構造は、低電圧で高電界を形成できて蛍光体を高輝度に発光させることができ、かつ蛍光体の発光特性を低下させることなく耐水性を付与できて発光特性が低下しにくい被覆蛍光体とすることができる利点を有している。なお各被覆膜の厚さは $30\mu\text{m}$ 以下、就中 $10\text{nm}\sim 1\mu\text{m}$ が一般である。

【0018】実施例1

厚さ $0.2\mu\text{m}$ のシリカ膜でコーティングした平均粒径 $20\mu\text{m}$ の ZnS (比較例2)に、 $\text{FSi}(\text{OC}_2\text{H}_5)_3$ を 50°C に加温しつつ発生させた蒸気としてアルゴンガスと共に供給する処理を室温で60分間続けてシリカ膜を

【0019】比較例1

平均粒径 $20\mu\text{m}$ の ZnS をそのまま用いた。

【0020】比較例2

室温 (25°C) 及び 5Torr の条件にて、減圧反応管内に設けたガラスフィルター上に平均粒径 $20\mu\text{m}$ の ZnS を 20g 保持し、それに $\text{Si}(\text{OC}_2\text{H}_5)_3$ を 50°C に加温して発生させた原料ガスをアルゴンガス ($73\text{cc}/\text{分}$) と共に反応管下部より ZnS 部に供給して流動層とすると共に、反応管の上部より酸素ガスを 50W 、1

3.56MHz の高周波を印加した高周波コイル域に $150\text{cc}/\text{分}$ の速度で供給してプラズマ化し、発生した酸素ラジカル、ないし酸素プラズマを流動層化した ZnS 部に供給する操作を約3時間続けて厚さ $0.2\mu\text{m}$ のシリカ膜でコーティングされた ZnS を得た。

【0021】評価試験

Si-OH吸収

実施例1又は比較例2で得た被覆 ZnS の赤外吸収スペクトルを調べ、 Si-OH に基づく吸収スペクトルを調べた。

【0022】輝度特性

厚さ $50\mu\text{m}$ のポリエステルフィルムからなるベース基板の片面に、銀粉含有の樹脂ペーストを部分塗布して幅 2mm の集電帯を形成後、 1TQ を分散含有させたフッ化ビニリデン系共重合体の酢酸セロソルブ溶液からなる透明導電塗料を塗布して厚さ約 $5\mu\text{m}$ の透明電極層 ($700\Omega/\square$) を形成し、その上にリード電極を付設後、実施例1又は比較例1、2で得た (シリカ被覆) ZnS を分散含有するフッ化ビニリデン系共重合体の酢酸セロソルブ溶液を塗布して厚さ約 $50\mu\text{m}$ の発光層を形成した。

【0023】他方、前記と同じ材質のベース基板の片面に銀粉含有の導電性塗料を塗布して厚さ約 $5\mu\text{m}$ の背面電極層を形成してリード電極を付設し、前記で得たベース基板と共にその層付設側を内側にして、チタン酸バリウム含有のフッ化ビニリデン系共重合体の酢酸セロソルブ溶液からなる厚さ約 $30\mu\text{m}$ の塗布層 (絶縁層を兼ねる接着層) を介して接着し、その接合体の上下に厚さ $100\mu\text{m}$ のポリエステルフィルム (PET) 又はポリクロロトリフルオロエチレンフィルム (PCTFE) を配置し、その周縁を接着して密封構造としEL発光体を得た。

【0024】前記のEL発光体の初期輝度 (駆動電圧: 100V) を測定後、それを 40°C 、 $90\%\text{RH}$ の雰囲気下、かつ 100V 、 400Hz による駆動状態下に放置し輝度が初期輝度の半分となる半減期を調べた。

【0025】前記の結果を表1に示した。なお輝度特性は、実施例1の場合を100としてその相対割合を示した。

【表1】



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Notes:

1. Untranslatable words are replaced with asterisks (****).
2. Texts in the figures are not translated and shown as it is.

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CLAIMS

[Claim(s)]

[Claim 1] In the silica membrane of the hydroxyl-group content to cover, the powder of a fluorescent substance General formula: F_nSiR_{4-n} (-- however, an alkyl group or an alkoxy group, and n of R are the integers of 1-3.) -- the manufacture method of the coat fluorescent substance characterized by diffusing the fluorine content organically silane expressed and carrying out a dehydration condensation reaction at silica membrane and low temperature.

DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Industrial Application] The coat fluorescent substance with which this invention was obtained about the manufacture method of the coat fluorescent substance which prevented degradation of a fluorescent substance is excellent in brightness, a luminescence life, and its maintenance nature, and can be preferably used for luminescence mold equipment, such as a lighting system and a display device, etc.

[0002]

[Description of the Prior Art] [the fluorescent substance used for formation of the light-emitting part in display devices, such as lighting systems, such as a fluorescent lamp and EL emitter, and an observation screen for electronic instruments, etc.] conventionally the cure covered with silica membrane since it deteriorates with moisture and the luminescence force and brightness fall is taken -- as the manufacture method of this coat fluorescent substance -- a CVD method and a sol -- the method of forming the silica-coding film by - gel method was known (JP,S61-23678,A).

[0003] However, when cooking temperature at the time of silica-coding film formation was made high, brightness etc. fell and the last twist which a fluorescent substance heat-deteriorates and is covered with coating membrane also had the trouble that a degradation rate also became quick and a life was shortened more. On the other hand, when the silica-

coding film was formed at 200-400 degrees C for heat deterioration prevention of a fluorescent substance, probably because it was inferior to waterproof nature, there was a trouble used as the coat fluorescent substance which deteriorates easily with moisture.

[0004]

[Problem(s) to be Solved by the Invention] Based on the knowledge acquired by having studied the scarce thing to it to waterproof nature that there is much content of a hydroxyl group and moisture permeates [therefore] easily if this invention is in the silica-coding film of said low-temperature formation It can form preventing degradation according the silica-coding film which there is little content of a hydroxyl group and is excellent in waterproof nature to the heat of a fluorescent substance etc., and let development of the manufacture method by which the coat fluorescent substance which is excellent in brightness, a luminescence life, or its maintenance nature is obtained be a technical problem.

[0005]

[Means for Solving the Problem] This invention the powder of a fluorescent substance in the silica membrane of the hydroxyl-group content to cover General formula: F_nSiR_{4-n} (-- however, an alkyl group or an alkoxy group, and n of R are the integers of 1-3.) -- the manufacture method of the coat fluorescent substance characterized by diffusing the fluorine content organicity silane expressed and carrying out a dehydration condensation reaction at silica membrane and low temperature is offered.

[0006]

[Function] By diffusing the fluorine content organicity silane expressed with said general formula in the silica membrane of hydroxyl-group content The fluorine content organicity silane carries out a dehydration condensation reaction at low temperature, such as a room temperature, through the hydroxyl group in silica membrane, and consumption clearance of the hydroxyl group in silica membrane is carried out, and the condensation reaction of silica membrane is promoted, and it reforms to precise silica membrane. And the silica membrane which reforms to the silica membrane of fluorine content and is excellent in water repellence is formed.

[0007] The silica-coding film which is excellent in waterproof nature can be formed without carrying out heat deterioration of the powder of a fluorescent substance, and the aforementioned result can obtain the coat fluorescent substance which is hard to produce moisture degradation and is excellent in brightness, a luminescence life, or its maintenance nature.

[0008]

[Example] the manufacture method of this invention -- general formula: F_nSiR_{4-n} (however, R -- an alkyl group or an alkoxy group --) n is the integer of 1-3. A coat fluorescent substance is obtained by diffusing the fluorine content organicity silane expressed in the silica membrane of the hydroxyl-group content which covers phosphor powder, and carrying out a dehydration condensation reaction at the silica membrane and low temperature.

[0009] The fluorine content organicity silane used in this invention should just show the reaction mechanism of dehydration condensation through a reaction with moisture or a hydroxyl group. [that the resultant based on an organic group (R) does not remain in silica membrane / thing / desirable] generally That whose R in said general formula is an alkoxy group like a methyl group, an ethyl group, a propyl group, the alkyl group like butyl or a methoxy group, an ethoxy radical, a propoxy group, and a butoxy radical is used.

[0010] A proper method may perform diffusion into the silica membrane of a fluorine content organicity silane. Although the method which supplies the air current of a fluorine content organicity silane to silica membrane through inert gas, such as helium and an argon, if needed is generally taken, the phosphor powder covered with silica membrane may be immersed in a liquefied fluorine content organicity silane

[0011] the temperature to which phosphor powder does not heat-deteriorate on the occasion of the dehydration condensation reaction of the fluorine content organicity silane diffused in silica membrane -- although you may generally heat at 200-400 degrees C, a desirable method is a reaction in low temperature, such as a room temperature.

[0012] There is no definition in particular about the silica membrane of the hydroxyl-group content which covers the phosphor powder of the object processed by a fluorine content organicity silane in this invention. Incidentally you may be formed with compound gestalten such as ZrO_2-SiO_2 besides [2] SiO_2 (for example, TiO_2-SiO_2).

[0013] Therefore, you may form the aforementioned silica membrane by a proper method Let silica membrane generally formed by the method which is easy to contain hydroxyl groups, such as a low-temperature CVD method in 400 degrees C or less, a reduced pressure CVD method, a plasma-CVD method, and a low-temperature sol gel method. be an object. Usually, 30 micrometers or less, although the thickness of the silica membrane of a processing object is 10nm - about 1 micrometer above all, it is not limited to this.

[0014] There is no definition in particular about the phosphor powder itself covered with silica membrane. Generally it consists of what activated zinc sulfide and cadmium-sulfide zinc by copper, manganese, aluminum, silver, chlorine, boron, etc., oxides, such as rare earth activation yttrium oxide, etc. Although the particle size of phosphor powder is arbitrary, based on mean particle diameter, 5-50 micrometers of 1 micrometers or more are common above all.

[0015] The phosphor powder which is used in this invention and which was covered with the silica membrane of hydroxyl-group content may be covered only with silica membrane by drawing 1 like instantiation, and may have other coating membrane more than one layer or two-layer inside silica membrane like instantiation in drawing 2. One in drawing is phosphor powder and the coating membrane of others [2 / 3 / silica membrane and].

[0016] Although the class of coating membrane other than the above mentioned silica membrane is arbitrary, the coating membrane which consists of a high dielectric as a thing effective in improvement in brightness is raised. The metallic-oxide system compound of transluency, etc. is raised with the high permittivity of Ta_2O_5 , aluminum $2O_3$, ZrO_2 , TiO_2 .

BaTiO₃, PbTiO₃, PZT (solid solution of PbZrO₃ and PbTiO₃), PLZT (La additive of PZT), and SrTiO₃ grade as an example of the high dielectric.

[0017] The coating structure which has the aforementioned high dielectric layer inside silica membrane has the advantage which can be used as the coat fluorescent substance with which a water resisting property can be given and a luminescent characteristic cannot fall easily, without being able to form a high electric field by a low battery, and being able to make a fluorescent substance emit light to high-intensity, and reducing the luminescent characteristic of a fluorescent substance. In addition, as for the thickness of each coating membrane, 10nm - 1 micrometer are general above all 30 micrometers or less.

[0018] The processing supplied to ZnS (comparative example 2) with a mean particle diameter of 20 micrometers coated with silica membrane with an example 1 thickness of 0.2 micrometer with argon gas as a steam generated warming FSi(OC₂H₅)₃ at 50 degrees C was continued for 60 minutes at the room temperature, and silica membrane was processed.

[0019] ZnS with comparative example 1 mean particle diameter of 20 micrometers was used as it was.

[0020] On condition of comparative example 2 room temperature (25 degrees C) and 5Toor, 20g of ZnS(s) with a mean particle diameter of 20 micrometers are held on the glass filter prepared in the reduced pressure coil. While supplying the material gas generated at it by warming Si (OC₂H₅)₄ at 50 degrees C to the ZnS section and using it as the fluid bed from the coil lower part with argon gas (a part for 73 cc/) From the upper part of a coil, supply oxygen gas to the high frequency coil region which impressed the RF (50W and 13.56MHz) the rate for 150 cc/, and it is plasma-ized. ZnS coating of the operation which supplies the generated oxygen radical or the ZnS section which fluid-bed-ized oxygen plasma was carried out by 0.2-micrometer-thick silica membrane continuously [ZnS / for about 3 hours] was obtained.

[0021] The infrared absorption spectrum of the coat ZnS obtained by the evaluation test Si-OH absorption example 1 or the comparative example 2 was investigated, and the absorption spectrum based on Si-OH was investigated.

[0022] [one side of the base substrate which consists of polyester film with a brightness property thickness of 50 micrometers] Carry out partial spreading and the resin paste of silver dust content After forming a 2-mm-wide current collection band, Apply the transparence conductive coating material which consists of a cellosolve acetate solution of the vinylidene fluoride system copolymer which carried out distributed content of the ITO, and a transparent electrode layer (700ohm/**) about 5 micrometers thick is formed. After attaching a lead electrode on it, the cellosolve acetate solution of the example 1 or the comparative example 1, and the vinylidene fluoride system copolymer that carries out distributed content of the ZnS (silica coat) obtained by 2 was applied, and the luminous layer about 50 micrometers thick was formed.

[0023] On the other hand, apply the conductive paint of silver dust content to one side of

the base substrate of the same construction material as the above, form a back plate layer about 5 micrometers thick, attach a lead electrode, and the layer attachment side is carried out inside with the base substrate obtained above. It pastes up through an application layer (glue line which serves as an insulating layer) with a thickness of about 30 micrometers which consists of a cellosolve acetate solution of the vinylidene fluoride system copolymer of barium titanate content, 100-micrometer-thick polyester film (PET) or a polychlorotrifluoroethylene resin film (PCTFE) has been arranged to the upper and lower sides of the zygote, the periphery was pasted up, it was considered as sealing structure and EL emitter was obtained.

[0024] The half-life when it is neglected at under the atmosphere of 40 degrees C and 90% RH and the actuation state by 100V and 400Hz, and brightness serves as half [of initial brightness] was investigated after measuring the initial brightness (driver voltage: 100V) of the aforementioned EL emitter.

[0025] The aforementioned result was shown in a table 1. In addition, the brightness was properly set the case of the example 1 to 100, and showed the relative rate.

[Table 1]

	Si-OH 吸収	初期 輝度	PET封止 輝度半減期	PCTFE封止 輝度半減期
実施例 1	なし	100	100	100
比較例 1	—	100	10	90
比較例 2	有り	100	≦10	60

[0026] The need for a closure film of according to the coat fluorescent substance by this invention being able to form EL emitter which is excellent in the endurance over humidity, and being hard to make the moisture like a polychlorotrifluoroethylene resin film penetrating from the outside, it is desirable to use a closure film which is different from the general-purpose films, such as easy polyester film.

[0027]

[Effect of the invention] According to this invention, the dry type structure is shown in Fig. 1, and the structure without varying our heat deterioration of the phosphor compound (Fig. 2) (the show-on Fig. 1 and 2 has the water repellence by a fluorine) for which is proper and is excellent in water-repellent nature can be formed. It is hard to produce moisture degradation, and the same coat fluorescent substance which is excellent in brightness, a luminescence film or its manufacturing nature can be obtained.

[Translation completed.]

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